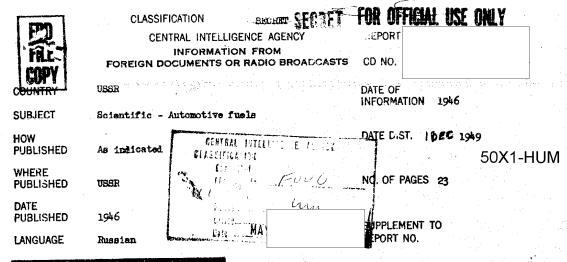
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RESEARCH ON USSR AVIATION GASOLINE

FOREWORD

/It has been noted that the Soviets' use of ethyl fluid does not conform to standards presently recognized in the United States.

Item I of this report contains frequent reference to component "R-9" and to tetracthyl lead with mention of a UB cthyl fluid similar to R-9, and with comparison figures and percentages for R-9 and the compounds used in the research project. Item II is presented as potential clarification on component "AK". Item III is the USER government standard specification for tetracthyl lead.

I. EXPERIMENT L RESEARCH ON LEAD REMOVER FOR ENGINEE

A. L. Feygin M. P. Alekseyeva

The work consisted of the following problems: (1) to develop a method of evaluating the removal of lead from the engine; (2) to analyze the effect of the concentration of brome and chloro derivatives on the effectiveness of lead removal; (3) to analyze the effects of brome and chloro derivatives of different chemical composition on removal of lead from the engine.

The material obtained should make it possible to draw mearer to the problem of selecting and classifying, by their effectiveness, combustion-chamber lead removers of various composition.

P. G. Kutikova and M. G. Tarasova participated in the physicochemical tests; A. A. Deryabin and E. V. Medvedev, in motor testing.

Deputy Chief of Talam, Frof M. M. Maslenikov, and to Engineer D. Ya. Kolomatskiy Department Chief No 9, assisted in the course of the work.

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INTRODUCTION:

A necessary property of organo-metallic antiknock compounds in operation should be the maximum possible elimination of metal from the engine cylinder. The problem of utilization of organometallic antiknock compounds is, therefore, closely connected with the problem of selecting the halogen-containing substances leading to the most complete conversion of the metal into halogen compounds easily carried off with the exhaust gares.

In research on halogen derivatives which convert lead into light, volatile compounds of lead, attempts were made first of all to use carbon tetrachloride. It proved to be unsuitable because its mixture with tetraethyl lead (hereafter referred to as TEL) is unstable. In storage it precipitates a white lead residue which lowers the effectiveness of its antiknock properties. (1)

Moreover, Midzhley tested as removers of lead such halogen derivatives as dichloroethane, chloro-tromoethane, chloroform, and others. Additional research and testing in the USSR was conducted on aircraft engines using dichloroethane as a lead remover.

At the present time, dibromoethane and ethyl bromide serve as lead removers, in the preparation of ethyl fluid on an industrial scale (ethyl fluids 1-T in UEA, and R-9 in UESR).

There is very little literature on experimental research in the field of lead removers, despite the fact that this subject is not only of theoretical, but also of practical interest.

The role of the halogen derivatives reduces to the conversion of metallic lead and lead oxide into a chloride or bromide which possesses high vapor tension and promotes the most complete possible removal of lead from the combustion chamber.

A comparison of the vapor tensions of halogen compounds of lead (2) shows that the condensation of lead chloride on the components of the combustion chamber begins with temperatures at which lead bromide is still completely removed. From this viewpoint, the chloride is less satisfactory than the bromide.

is greater or smaller capacity of various halogen derivatives toward thermal decomposition accompanied by separation of the halogen has great significance under engine conditions since there is no doubt that the presence of the free halogen at the moment of decomposition of the tetraethyl lead in the engine is the condition which the lead removal process is based upon. (At atmospheric pressure this disintegration takes place at 275 degrees centigrade.) (4)

Inasmuch as it was not possible for us to discover in available literature works on the reaction of lead compounds with organic halogen derivatives, we have developed the very interesting investigations of Hartel, Meer, and Polany (3) on the speed of reaction of alkyl chlorides with sodium vapors.

The authors found the following rules:

- (1) The speed of the reaction increases in the order: fluorine--chlorine--browine--iolins (for both alighatic and aromatic compounds).
- (2) The speed of the reaction increases with the increase in the length of the hydrocarbon chain.
- (3) Tertiary compounds favor reaction more than do secondary or primary compounds.
- (4) The splitting up of the chain has negligible influence on the speed of the reaction.

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- (5) Compounds with double honds of the atom to which the chlorine is attached are less favorable to reaction than the corresponding saturated compounds.
- (6) Compounds with double bonds on the carbon atom adjoining the one to which the chlorine is attached are more favorable to reaction.
 - (7) The presence of the carbonyl group increases the speed of the reaction.
- (8) The speed of the reaction increases with an increase in the number of chlorine atoms in the molecule, and the closer the carbon atoms, to which the halogens are attached, are grouped together, the more conducive are the compounds to reaction.

The discovery by Polany and associates of the rules for the reaction of scdium vapor with various halogen compounds arouses great interest. It is possible that the rules obtained for metallic sodium will also held good for lead.

We did not find any works in which the thermal stability of a series of halogen compounds would be equal with the same measuring methods and the same temperature and pressure. Nevertheless, it is possible to obtain, from the data brought forth here, certain ideas on the behavior of organic halogen compounds in the engine. Thus, from Nef's data (5), ethyl bromide under atmospheric pressure does not decompose up to 425 degrees centigrade.

Ethyl bromide begins to decompose at h80 - 525 degrees centigrade. At 500 - 600 degrees centigrade, it decomposes completely into hydrogen bromide and athylene. As an intermediate stage in this decomposition, Nef assumes the formation of a free radical of ethylidene which by regrouping gives ethylene:

$$C_2H_5H_7 \longrightarrow CH_3CH_2 + HH_7$$
 $CH_3CH = \longrightarrow C_2H_4$.

Vernon and Daniels (6) consider that ethyl broade during heating directly undergoes monomolecular grouping. According to Sokowin's data (8), dibromosthe when heated to 300 - 315° in a closed tube results in a system of equilibrium:

The author found that the following reactions are possible in this system:

- (1) C2H1Br2+HBr 2 C2H5Br +Br2
- (2) CoHabra+Bra CoHaBra+HBr

Products containing in their molecules a larger number of halogen atoms begin to decompose at a lower temperature.

Thus, tetrabromoethane, according to Sabanejew's data (9), decomposes with heating at 190 degrees centigrade and forms tribromoethane and hydrogen-bromide.

Chlorine compounds, as with the browine ones, at temperature of decomposition form hydrogen chloride and wasaturated compounds. Thus, dichloroethane, at temperatures above 400 degrees centigrade (10) gives HCl and winyl chloride:

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Ivanov and Kreyn (11) studied the oxidizing decomposition of dichloroethene during its combustion in a horizontal pipe furnace. It was established that during the burning of pure dichloroethene (as a mixture with various fuels) all the chlorine contained therein separates in the form of hydrogen chloride.

In the products of the reaction-free chlorine, its oxide derivatives and phosgene were not found.

Compounds which contain three or more halogen atoms, during thermal decomposition, give off free halogens in addition to unsaturated compounds and hydrogen halides.

Thus, when chloroform is heated in a cylinder to 425 degrees centigrade under a pressure of 20 atmospheres the products of decomposition are chlorine, hydrogen chloride, hexachloroethane, and others. When carbon tetrachloride is heated in an incandescent tube (13), chlorine: tetrachloroethylene and hexachloroethane are formed. Aromatic compounds with he logens in the nucleus are more heat-stable than helogen-containing substances of the alignatic series.

Meyer and Hofman (14) established that chlorine and bromine, when replacing the hydrogen of a benzene nucleus, make this nucleus more stable. Thus, the lower decomposition temperature of benzene is equal to 650 degrees, while halogen derivatives of benzene and toluene (icdo-,bromo-, chloro-, and fluoro-) decompose at a higher temperature--that is, 700 - 800 degrees centigrade. It was also established that, of the halogens, icdine breaks off the most readily, followed by bromine, chlorine, and most difficult of all, fluorine.

In aromatic halogen compounds with the halogens in side chains, and in simpler derivatives the separation of the halogenes (appearing as hydrogen halides) takes place at comparatively lower temperatures then in compounds with the halogens in the nucleus.

Data regarding thermal decomposition of certain other organic halogen compounds are also brought forth in the following works (5-10, 12-23). From these works it was seen that thermal stability was lowest in iodo derivatives, followed by bromo derivatives, and chloro derivatives, and, in addition that polyhalides were less stable than monohalides.

In a short review of the conditions of thermal stability of various organic halogen derivatives, it follows that, to all appearances, the best lead removers will be the substances which are more inclined to thermal decomposition, with a release of halogen or hydrogen halide, whereupon the formation of the halide must coincide in time as closely as possible with the moment of decomposition of the TEL and deposit of the atomic lead in the engine.

A. Objects and Methods of Research

For the investigations there were taken TEL, gasoline E-78, and a series of halogen carriers: (1) ethyl bromide, (2) dibromoethane. (3) tetrabromoethane, (4) dichloroethane, (5) chloroform, (6) carbon tetrachloride, (7) chlorobenzene, (8) a mixture of ortho- and para-dichlorobenzenes, (9) bromobenzene, (10) para-chlorobenzene, (11) d-monochloronaphthalene.

Commercial tetrasthyl lead had a specific gravity of 1.58 at 20 degrees centigrade and contained 98.05 percent TEL by weight. The majority of the lead removers were obtained from special factories, and before the experiments, were distilled and desiccated over calcium chloride. The bromobenzene, para-chlorobromobenzene, and tetrabromoethane were synthesized by us.

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The gasoline B-78 was standard, with a specific gravity of 0.735 at 20 degrees centigrade, initial boiling point at 65 degrees centigrade and final boiling point at 170 degrees centigrade. The gasoline consisted of 8.4 percent aromatics, 26.6 percent naphthene, and 65.5 percent paraffin hydrocarbons. In Table I there is a compilation of physicochemical properties of the halogen carriers used by us in the experiments.

The quantitative determination of lead in the commercial TEL and ethyl fluid was carried out by the standard molybdate method. The quantitative determination of the total lead deposit (helogen, oxide, and metallic) was carried out by the method developed in the Military-Air Academy KA.

Table I. Physicochemical Properties of Halogen Carriers

| No | Compounds | Formulas | Boiling Point | Specific Gravity (d20) | Halogen | Halogen (theoretical %) | Solidifi- cation Point (°C) |
|-----|---|--|----------------------------------|------------------------------|---------|------------------------------|--------------------------------------|
| . 1 | ethyl bromide | C ₂ H ₅ Br | 38-39.5 | 1.44 | 72.50 | 73.25 | |
| 2 | dibromoethane | CH ₂ BrCH ₂ Br | 129-132 | 2.18 | 85.70 | 85.08 | • e ^v |
| 3 | tetrabromoethane | CHBr ₂ CHBr ₂ | 190 with decomposition | 2.94 | 91.76 | 92.47 | -17 |
| 4 | dichleroethane | CH ² C1CH ² C1 | 83-85 _. | 1.254 | 72.00 | 71.7 | |
| 5 | chloroform | CHC13 | 61-62.5 | 1.476 | 88.80 | 89.13 | |
| 6 | carbon tetrachloride | cci [‡] | 76.5-77.5 | 1.593 | 91.90 | 92.20 | |
| 7 | chlorobenzene | C6H5C1 | 130-134 | 1.105 | 30.00 | 31.5 | 55 |
| 8 | mixture of ortho- and para-dichloro- benzene. | Waste products during benzens chlorination | Initial BP -90 Final BP -194 | 1.257 | 44.80 | | 0 |
| 9 | bromebenzene | C ₆ H ₅ Br | 154-155.5 | 1.492 | 50.40 | 50.90 | -27 |
| 10 | pera-chlorobenzene | Central Collection | | | 60.00 | bromo \$1.75 chloro 18.52 | fusion temp 63.3 |
| 11 | X-monochloro naphthalene | C ₁₀ H ₇ C1 | Initial EF -250 Final EP -297 | 1.215 | | | |

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Removal of Lead From the Combustion Chamber of the Engine

To evaluate the effectiveness of halogen carriers as load removers it was necessary to develop a laboratory method which would approach the actual operating conditions of a contemporary aircraft engine. Such a method did not exist at the beginning of this work. The very simple and convenient method developed by M. B. Neyman (2) for a comparative evaluation of lead removers did not satisfy fully the following conditions established by us:

- (1) The method must approach as close as possible to the operating conditions of fuel consumption in modern aircraft engines.
- (2) The method must be as accurate as possible, convenient and must not require a large amount of material or time.
- (3) The amount of deposit produced should be sufficient for a complete analysis and evaluation of the halogen carriers of various chemical compositions as lead removers.

For the solution of the given questions we used a one-cylinder Waukesha engine (the army method for determining octane number was applied), in which it was possible to perform an evaluation of the degree of lead plating and lead formation in such responsible perts of the engine as the exhaust valve, spark plugs, etc.

The evaluation of the removal of lead from the combustion chamber of the engine was performed by us as follows:

A determined quantity of gasoline was primed with the experimental ethyl fluid consisting of TEL and halogen carriers. The experimental ethyl fluid was prepared by mixing tetraethyl lead and halogen carriers of various chemical composition, taken in aclecular proportions. For example, two wol of ethyl bromide were taken with one mol of tetraethyl lead, and in the case of dibromoethane-one mol. Thus in both cases, the proportion was one mol of halogen for one mol of TEL. The composition of the TE and halogens was determined analytically both for the original components and in prepared fuel. The gasoline, primed with the experimental fluids, was burned in the engine unit (without stopping the engine) at a determined rate. After this, the deposit was taken off all parts of the combustion chamber, weighed and analyzed as tototal quantity of lead, lead halogen, lead oxide, and metallic lead. On the basis of these data, the percentage of lead taken out of the combustion chamber during the engine operation on this or another sample of experimental fluid was calculated.

To shorten the duration of the experiments, and to accumulate an adequate amount of deposit and lead in the combustion chamber necessary to permit an analysis, it was decided to prepare the gasoline in a quantity of 10 kilograms with a high concentration of ethyl fluid (1. milliliters per kilogram of fuel). However, it was not clear whether the data obtained with gasoline containing a high concentration of ethyl fluid could be applied to aircraft engine operation where, as is known, the fuel contains 4 milliliters per kilogram of fuel. For clarification of these and other questions posed above, we carried out a series of experiments on the one-cylinder engine selected by us.

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Table II. Influence of the Concentration of Ethyl Fluid in Gasoline and Duration of Engine Operation on Rate of Deposit Formation and Deposition of Lead in Combustion Chamber of Engine

| 30 . | Composition of the Antiknock Compound (TEL) | Quantity of gesoline used in the experiment (kg) | Contents of experimental fluid per kg of fuel, converted to R-9 (ml) | Lead content (gr/kg of fuel) | each r | | Inteke valve | |
|-------------|---|--|--|------------------------------|---------------|------|--------------|------|
| 1 | TEL (without remover) | 10 | 10 | 5.28 | 0.41 | 3.21 | 0.05 | 2.66 |
| 2 | TEL (1 mol)+ ethyl bromide (2 mol) | 9.6 | 9.5 | 5.03 | 0.31 | 0.36 | 0.01 | 2.13 |
| 3 | TEL (1 mol) + ethyl browide (2 mol) | 10 | 10.15 | 5.33 | 0.50 | 0.40 | 0.15 | 1.84 |
| ħ. | TEL (1 mol) + dibromo- ethane (2 mol) | 25 | 4.05 | 2.14 | 0.07 | 0.91 | 0.20 | 0.85 |
| 5 | TEL (1 mol) + dibromo- ethene (1 mol) | 10 | 9.5 | 5.01 | O. 3lt | 0.36 | 0.45 | 1.40 |
| 160 | TEL (1 mol) + dibromo- ethane (1 mol) | 10 | 9-93 | 5.21 | o .3 6 | 0.47 | 0.40 | 1.48 |
| 7 | TEL (1 mol) + dibromo- ethane (2 mol) | 25 | 4.2 | 2.25 | 0.07 | 0.41 | 0.17 | 1.05 |

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Table II (Contd)

| | | | | 1 | | | |
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| .No. | Composition of the Antiknock Compound (MEL) | Weight of deposit in the combination chamber (gr) | Lead content of deposit in combustion chember (β) | quantity of lead introduced with gasoline during run (gr) | Quantity of lead discovered in combistion chamber after run (gr) | Lead deposited in combustion obsmber (%) | Lead removed (\$) |
| 1 | TEL (without remover) | 6.34 | 84.35 | 52.8 | 5•3 ^k | 10.1 | 89.98 |
| 2 | TEL (1 mol) + ethyl bromide (2 mol) | 2.60 | 54.4 | 48.26 | 1.46 | 3.2 | 96.8 |
| 3 | TEL (1 mol) +othyl bromide (2 mol) | 2.89 | 54.8 | 53 ·3 | 1.58 | 2.97 | 97.03 |
| 4 | TEL (1 mol) + dibromo- ethane (2 mol) | 2.03 | 69.14 | 5 3. 50 | 1.40 | 2.61 | 97.39 |
| 5 | TEL (1 mol) + dibromo- ethane (1 mol) | 2.50 | 55.58 | 50.1 | 1.41 | 2.81 | 97.19 |
| 6 | TEL (1 mol) + dibromo- ethane (1 mol) | 2.71 | 57.5 | 52.1 | 1.56 | 3.00 | 97.00 |
| 7 | TEL (1 mol) + dibromo- ethane (1 mol) | 1.70 | 63.0 | 55.5 | 1.07 | 1.93 | 98.17 |

Table II shows the results of the experiments with gasoline B-78 containing various amounts of TEL with ethyl bromide and dibromoethane as lead removers.

One sample of gasoline contained pure TEL (without a remover) in the amount of 3 millimeters per kilogram of fuel. Four samples of the gasoline contained a quantity of TEL (5 milliliters) which in lead content was equivalent to 10 milliliters of standard ethyl fluid (R-9). The amount of gasoline used was 10 kilograms. Two samples of gasoline contained 2 cubic milliliters TEL which was equivalent to 4 cubic milliliters of standard ethyl fluid R-9, and 25 kilograms of fuel was used for each run.

In this way the total amount of lead introduced into the combustion chamber of the engine was the same for all runs. Table II shows fairly well the convergence (similarity) of the parallel experiments. Thus, the total amount of deposit and percent of the lead deposited in the combustion chamber using ethyl bromide equalled respectively 2.60-2.89 grams and 3.2-2.97 percent.

The samples of gasoline containing TEL and halogen carriers showed, in comparison with gasolines with TEL but without halogen carriers, considerably better results in regard to removal of lead from the combustion chamber of the engine. Thus, the lead removed amounted to about 90 percent for fuel without removers and attains 90% with the presence of the halogen carriers. These data distinctly show the effective role of halogens as lead removers.

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It follows from the table that the concentration of ethyl fluid in the gasoline (10 and 4 milliliters R-9 per kilogram fuel), under the condition that the same amount of lead was introduced into the combustion chamber, does not in practice influer to the percent of lead removed from the combustion chamber. Thus, the lead removed in these experiments constituted around 97%, and the total percentage of lead in the deposits varied within 55-70 percent.

These data show that for a comparative study of the effectiveness of various lead removers it is possible to set a limit of 10 hours engine operation on gasoline containing approximately 10 milliliters ethyl fluid per kilogram fuel in order to obtain a sufficient amount of deposit for antitytical purposes. At the same time, it is possible to obtain, with an economical expenditure of time and materials, approximately the same results as in operations utilizing gasolines containing the standard amount of ethyl fluid (4 milliliters) and used in modern engines.

On the basis of the preliminary experiments carried out, a method was developed for a comparative evaluation of fuels, with the addition of TEL and various halogens, in regard to deposit formation and removal of lead, approximating the operating conditions of aircrait engines.

It is obvious that such procedure can be applied not only to the onscylinder unit selected by us, which was designed for the determination of the octane number of fuels according to the army method, but also to other Waukeshe units and other motors, with the application of other methods for evaluating the antiknock properties of fuels (motor, etc.). For this research it is also possible to use a motorcycle engine successfully. With such engines, and utilizing the method developed by us, it is possible to carry out a comparative evaluation not only for tetraethyl lead but also for other antiknock compounds.

The experimental work performed by us in regard to the influence of the chemical composition of the halogen carriers on the removal of lead was done with a one-cylinder unit under the following whitions:

Engine Data

Cylinder diameter Piston travel

Spark advance

| | Engine Operating Conditions | | | | | | |
|-----------|----------------------------------|-------------------|--|--|--|--|--|
| 1. | Number of revolutions | 1200 rpm | | | | | |
| 2. | Compression ratio | 6.5 | | | | | |
| 3. | Temperature of the engine jacket | 100°C | | | | | |
| 4. | Mixture | 0.85 | | | | | |
| 5. | Coolant | water | | | | | |
| 6. | Temperature of the suction air | 20-25°C | | | | | |
| 7. | Oil temperature | 75 ⁰ C | | | | | |
| 8. | Amount of fuel expended on a run | 10 kg | | | | | |
| 9. | Duration of each run | 10 hours | | | | | |
| 10. | Hourly fuel consumption | 1 kg | | | | | |
| 11. | Hourly oil consumption - 9 - | 10-15 | | | | | |

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- 12. Amount of antiknock compound consisted approximately of 5 ml of TEL/kg fuel, which corresponded to 10 ml of ethyl fluid (R-9).
- 13. One grade of oil was used in all runs--a mixture of 70% MZS cil+30% transformer oil with a viscosity of 6.4°E at 50°C. Viscosity of the waste oil in all cases was within the limits 6.2-6.4°E.
- C. Influence of Chemical Composition of Brome Derivatives of the Aliphatic Serjes on Removal of Lead From the Combustion Chamber of the Engine
- 1. The Influence of Dibromoethane Concentration in Ethyl Fluid on the Removel of Lead From the Combustion Chamber of the Engine.

The influence of halogen carrier concentration in ethyl fluid on removal of lead from the combustion chamber of the engine was studied for dibromosthams (DEE) -- a component of the standard antiknock compound -- ethyl fluid.

Figures 1-3 show the change in lead deposit in the combustion chamber of the engine, deposit formation on a most vital part of the motor--the exhaust valve, percontage of lead in the deposit, and also the change in the chemical ecosposition of the deposit, that is, the percent of lead helogen, lead oxide, and metallic lead, depending on the dibromoethane concentration in the ethyl fluid.

Figure 1 shows the point-by-point decrease of the weight of the deposit on the exhaust valve from 3.22 grams with complete absence of halogen carriers to 0.20 grams with 7.5 centimoles of dibromosthane for 2.5 centimoles TEL in a kilogram of fuel. The greatest decrease of deposit formation takes place when 2.5 centimoles of dibromosthane is introduced. In this case, the amount of deposit paresses from 3.22 to 0.5 grams.

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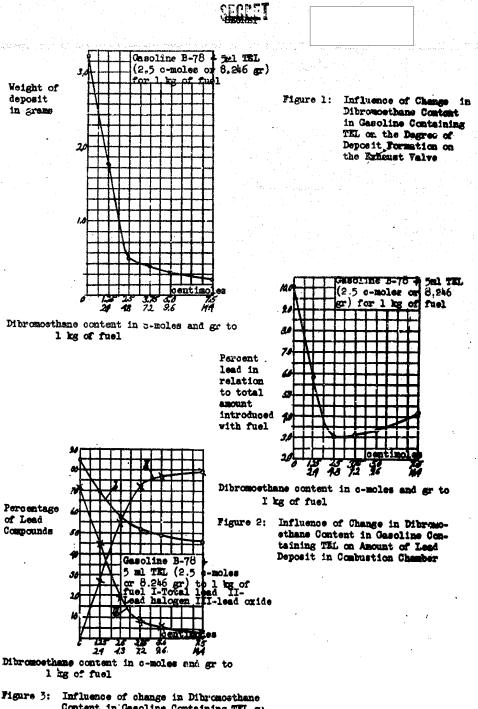


Figure 3: Influence of change in Dibromoethane Content in Gasoline Containing TEL on Amount of Total Lead, Lead Halogen, and Lead Oxide in Deposit

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The additional introduction of dibromoethane up to 7.5 centimoles was reflected only to a small extent in the change of deposit formation on the valve.

Although, deposit formation on the valve in the range from 3.5 to 7.5 centimoles is practically constant, the total amount of deposit in the combustion chamber with the maximum concentration of dibromoethane (7.5 centimoles) is not less but even more than for lower concentrations.

The increase in the total amount of deposit results from an accumulation on the cylinder head of the lead halogen which settles on the cooler surface at the same time that the lead halogen volatilizes from the surface of the much hotter exhaust valve. It is possible that the lead halogen in the presence of a surplus amount of the halogen enters into reaction with the organic substances of the deposit, giving off a metallo-organic compound which settles on a cooler spot in the engine, carbonizing the deposit and leading to an increase in the accumulation of deposit on the cylinder head.

Rules similar to those for carbon formation apply to lead deposit in the combustion chamber of the engine, depending on the dibromoethane concentration in the ethyl fluid.

Figure 2 shows that 10.1 percent of the lead was deposited in the combustion chamber during operation with ethyl fluid minus dibromoethane. In proportion to the increase of dibromoethane the amount of lead deposited in the combustion chamber decreases. The most during decrease in lead deposition takes place with 2.5 centimoles of dibromoethane with from 2.5 to 5 centimoles there is hardly any effect on the change in the amount of lead deposited in the combustion chamber.

However, when the concentration of diffremoethane was increased from 5 to 7.5 centimoles, lead deposition not only failed to decrease but in fact increased from 3.30 to 4.10 percent. This same phenomenon also took place during study of deposit formation in the combustion chamber of the engine.

It is evident from Figure 3 that the percent of total lead in the deposit falls off sharply from 84.35 to 57.5 percent during the transition from pure TEL to ethyl fluid containing 2.5 centimoles of dibromoethans. With a further increase in dibromoethans concentration to 7.5 centimoles, the percent of lead in deposit changes very little (from 57.5 to 46 percent). While the total percentage of lead in the deposit continues to remain more or less constant independent of DBE concentration in the ethyl fluid, the qualitative characteristic of the lead compounds undergoes a remarkable change in response to helogen carrier concentration.

The lead halogen content in deposit increases sharply with increase in LHE concentration. The lead halogen content increased from 0.0 percent (pure TEL) to 55 percent (2.5 centimoles TEL to 2.5 centimoles DHE). The lead halogen content in deposit increases (from 57.5 to 72.5%) with an increase in DHE concentration from 2.5 to 3.75 centimoles. Still further, the lead halogen content thanges very little with an additional increase in DHE concentration (from 72.4 to 79 percent).

The lead oxide centent in the deposit changes conversely with the above pattern for lead halogen. The percentage of lead oxide falls from 72.4 (pure TEL) to 8.01 percent (2.5 centimoles TEL+3.75 centimoles (DRE). The percentage of satallic lead in the deposit was not very large and varied from 0 to 17.0 percent. With 7.5 centimoles of dibromoethane the metallic lead disappears completely. All these data show clearly that the engine operation on gasoline with pure TEL without addition of halogen carriers results in excessive deposit of lead in the combustion chamber (10.1 percent).

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With such a deposit of lead the functioning of spark plugs, valves and other parts of the engine becomes impossible and the engine breaks down. By introducing dibromoethane halogen carrier the removal of lead from the combustion chamber improves considerably increasing in proportion to the addition of dibromoethane.

Completely reliable engine operation from the viewpoint of deposit formation on the exhaust valve can be arrived at by including in one kilogram of fuel approximately 3.75 centimoles of dibromoethane to 2.5 centimoles of TEL.

 Effect of Chemical Composition of Bromo-Containing Substances of the Aliphatic Series on Removal of Lead From the Combustion Chamber of the Engine

Research on the influence of successive bromine substitution of hydrogen atoms in hydrocarbon molecules on the removal of lead from the combustion chamber was studied with ethyl bromide, dibromoethane, and tetrabromoethane.

Figures 4 - 6 show the change in lead deposition and deposit formation in the combustion chamber of the engine, and also the change of the chancel composition of the deposit, relative to the successive substitution of bromine for the hydrogen atoms of the ethane molecules. It follows from Figures 4 - 6 that the quantity of lead (in percent of the given load) deposited in the combustion chamber decreases in proportion to the bromine substitution of the ethane hydrogen atoms. Thus, the lead obtained was, in the case of ethyl bromide 2.98 percent, dibromosthane 2.41 percent, and tetrabromosthane 2.12 percent. It follows from these data that the sharper drop in lead deposition takes place with the change from ethyl bromide to dibromosthane.

Further bromine substitution to tetrahromoethane decreases the amount of lead deposited but not to the same degree as in the first case. Accordingly the deposit formation in the combustion chamber varies during operation with the above-mentioned halogen carriers.

In this way, ethyl bromide, in relation to removal of lead from the combustion chamber, yields to dibromoethane and tetrabromoethane. This is also confirmed by the chemical composition of the deposits. Thus the lead halogen content while operating on ethyl bromide is less (49.3 and 58 percent), and lead content more (24.8 and 21.5 percent) than while operating on dibromoethane.

The total percentage of lead in the deposit in the case of all halogen carriers investigated was about the same (52.5-58 percent).

The experimental results infirm the fact that thermal stability decreases in this order or the halogen carriers: ethyl bromide, dibromoethane, and tetra-bromoethane.

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Percent Lead of Total Amount Introduced With Fuel

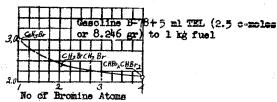


Figure 4: Influence of Degree of Substitution of Hydrogen Atoms in Halogen Carriers by Bromine on Extent of Removal of Lead from Combustion Chamber of Engine.

Weight of Deposit in Grams

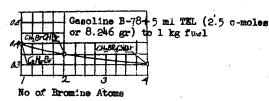


Figure 5: Influence of Degree of Substitution of Hydrogen Atoms in Halogen Carriers by Bromine on Deposit Formation on Exhaust Valve

Lead Compound Contents in Percent

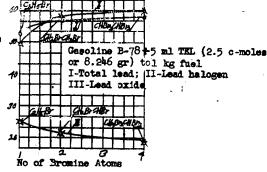


Figure 6: Influence of Degree of Substitution of Hydrogen Atoms in Halogen Carriers by Browins on Amount of Deposit of Total Lead, Lead Halogen, and Lead Oxide.

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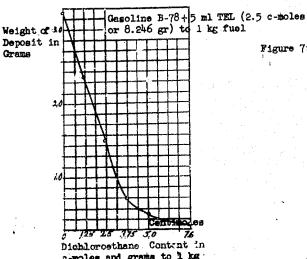
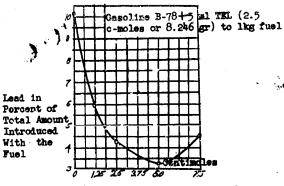


Figure 7:

Influence of Change in Dichloroethane Content of Gasoline Containing TEL on Extent of Deposit Formation on Exhaust Valve.

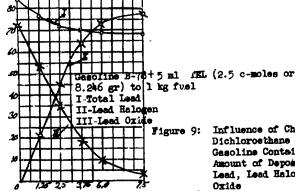
o-moles and grams to 1 kg of fuel Influence of Change in Figure 8:

Dichlorosthans Content of Gasoline Containing TEL, on Extent of Lead Deposit in the Combustion Chamber"



Dichloroethans Content in c-moles and gr to 1 kg of Fuel

Percentage of Lead Compounds



Influence of Change in Dichloroethane Content of Gasoline Containing TEL on Amount of Deposit of Total Lead, Lead Halogen, and Lead Oxide

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- D. Influence of Chemical Composition of Chloro Derivatives of the Aliphatic Series on kemoval of Lead From the Combustion Chamber of the Engine
 - Influence of Dichloroethane Concentration and Thyl Fluid on Removal
 of Lead From the Communition Chamber of the Engine.

The influence of chloro-carrier concentration in the ethyl fluid on removal of lead from the combustion chamber of the engine was studied with dichlorosthans (DCE) which in its themical structure is similar to dibromoethane, a component of the widely used ethyl fluid.

Figures 7 - 9 show the change in lead deposit in the combustion chamber of the engine, deposit formation on a most vital part of the engine--the exhaust valve, percentage content of lead in deposit, and also the change in the chamical composition of the deposit, i.e., its lead halogen, lead oxide and metallic lead content, relative to dichlorosthane concentration in the ethyl fluid.

Figure 7 shows the step-by-step decrease of the weight of the deposit on this exhaust valve from 3.22 grams for total absence of dichlorosthams to 0.36 grams for 7.5 centimoles of dichlorosthams. The greatest decrease of deposit formation takes place with the introduction of 3.75 centimoles of dichlorosthams. In this case, the amount of deposit decreases from 3.22 to 0.58 grams. Further introduction of dichlorosthams to 7.5 centimoles gives a much smaller effect, at which time the deposit decreases from 0.68 to 0.36 grams. It should be noted that although the deposit formation on the exhaust valve for 7.5 centimoles is somewhat smaller than for 5 centimoles of dichlorosthame, nevertheless the amount of deposit in the combustion chamber for 7.5 centimoles turns out to be not smaller but larger than Tor 5 centimoles. The increase in total amount of deposit results as a consequence of deposition in the cylinder head. This increase, as in the case of dibromosthame, can be explained by the accumulation in the cylinder head halogen which settles on the cooler surface at the same time that the lead halogen volatilizes from the surface of the hotter exhaust valve.

Rules similar to those in carbon formation apply to lead deposition in the combustion chamber of the engine, depending on the dichloroethane concentration in the ethyl fluid.

It follows from Figure 8 that 10.1 percent of the lead is deposited in the combustion chamber while operating v'thout dichloroethans. In proportion to the addition of dichloroethane, the amount of lead deposited in the combustion chamber decreties. The sharpest decrease in deposit of lead is for 3.75 centimoles of dichloroethane (10.1 and 3.6 percent). Further addition of dichloroethane does not have as strong an effect. The percentage of lead deposited in the combustion chamber falls from 3.6 to 3.25 while the dichloroethane content increases from 3.75 to 5 centimoles to one kilogram of fuel. With further addition of DCE to 7.5 centimoles, the amount of lead deposited no longer decreases but rather increases to 4.54 percent. This phenomenon also takes place during deposit formation in the combustion chamber of the engine.

It follows from Figure 9 that the percent of total lead (curve I) in deposit changes slightly as the dichloroethane concentration is increased. The percent of lead falls from S4.35 to 70 with the variation from pure TEL to ethyl fluid containing 3.75 centimoles of DCE, after which the percentage of lead in deposit continues to remain practically constant, regardless of the amount of DCE alded.

Although the total percentage of lead in deposit continues to remain more or less constant, the quantitative character of the lead compounds undergoes a considerable variation, dependent upon the concentration of halogen carrier. Thus, the lead halogen increases abruptly with an increase of DCE concentration (Figure 9). The lead halogen content increased from 0.0 (pure TEL) to 64 percent (3.75 centimoles DCE). Conversely, the lead oxide content in deposit decreases.

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On the basis of the data here, it can be stated that dichloroethane improves lead removal from the combustion chamber, with the effectiveness increasing in proportion to the addition of dichloroethane. If we judge from the viewpoint of the exhaust valve, then the minimum deposit formation takes place at 7.5 centimoles of dichloroethane to 1 kilogram of fuel.

Engine operation is entirely satisfactory with this degree of deposit formation.

Influence of the Chemical Composition of Chlore-Containing Substances
of the Aliphatic Series on Removal of Lead From the Combustion Chamber
of the Engine.

A comperison of chloro derivatives of the aliphatic series was carried out with Atchloroethane, chloroform, and carbon tetrachloride.

Table III gives the chemical composition of the corresponding deposits. Table III (average values) shows very similar data for deposit formation on the exhaust valve and for lead deposit in the combustion chamber while operating the engine on gasoline with addition of TEL and halogen carriers—dichloroethane, chloroform, and carbon tetrachloride.

The weight of deposit on the exhaust valve for all the halogen carriers listed in found within the limits 1.5 - 1.55 grams, and the lead content of the deposit in the combustion chamber varies in the limits 3.7 - 4.0 percent. However, all of the relogen carriers listed are inferior in operation to dibromoethane.

Table III. COMPOSITION OF THE DEPOSIT

| Composition of Antiknock Compound | Weight of Deposit on Exhaust Valve (gr) | Quantity of Lead in the Combustion Chamber (gr) | Lead Peposited in the Combus- tion Chamber (\$) | Toud Removed (\$) |
|--|---|---|---|-------------------|
| TEL (1 mol) + dibrom ethane (1 mol) | 0.47 | 1.56 | 3,50 | 97.00 |
| TEL (1 mol)+ dichlor ethane (1 mol) | 1.48 | 2.23 | 4.25 | 95.75 |
| TEL (1: >1) + chloro (2/3 mol) | 1.56 | 2.08 | 3.99 | 96.01 |
| TEL (1 mol) + carbon tetrachloride (1 | /2 mol) 1.56 | 1.96 | 3.74 | 96.26 |

Thus, while operating on dibromoethane, deposit formation on the exhaust valve is one-third as much as with dichloroethane, chloroform, and cartom tetrachloride. Thus, the antitative indexes for all the listed chloro-containing substances appeared practically the same when used in the one-cylinder engine.

E. Influence of Halogen Derivatives of the Aromatic Series on Removal of Lead From the Combustion Chamber of the Engine

The influence of halogen-carriers of the arcmatic series on removal of lead from the combustion chamber of the engine was studied with bromobenzene, chlorobenzene, a mixture of ortho-and para-dichlorobenzenes, and chloromephthalene. Besides this, for investigating the effectiveness of the presence in the benzene micleus of two halogens, bromine and chlorine, on the removal of lead, para-chlorobromobenzene was also tested against chloro- and bromobenzene.

The data in Table IV show that, in regard to amount of lead (average data) deposited in the combustion chamber and deposit formation on the exhaust valve, the halogen-carriers investigated are arranged in the following order:

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Table IV

| | Lead Depos Chamber of | | Combustion (%) | Formed on t |
|--------------------|--------------------------|------|----------------|-------------|
| Dibromoethane | | 2.41 | | 0.35 |
| Chlor-bromobenzene | 100 6 62 4 | 3.14 | | 1.06 |
| Chloronaphthalene | | 4.04 | | 1.30 |
| Bromobergene | | 4.23 | | 1.27 |
| Dichlorebenzene | | 4.86 | | 1.63 |
| Chlorobenzene | | 5.11 | | 1.86 |

The percentage of total lead in deposit is smallest in the case of chloro-bromobenzene (64.18 percent on the average). For all the remaining halogen carriers the total lead content varies from 69 - 76.8 percent.

The lead halogen content in deposit shows up largest in the case of chloronambthalene. For the remaining halogen carriers the percentage of lead halogen is nearly the same. The percentage of lead oxide varies in accordance with the same pattern.

From the data here, it follows that all the halogen carriers of the aromatic series were seen to be less effective than dibromoethene--the halogen carrier of standard ethyl fluid.

Thus, in relation to deposit formation on the enhaust valve, the tested halogen carriers are less effective than the dibromoetheme.

As a lead remover, of -monochloromaphthalene proved to be a fairly effective, although inferior to dibromoethane. Chloromaphthalene is approximately 20 percent more effective than chlorobenzene, and is practically equivalent to bromobenzene.

Experimental data show that the benzene nucleus with two halogens in the nucleus—chlorine and bromine—with respect to lead removal, proves to be somewhat more effective than the chlorobenzene or bromobenzene separately.

The experimental data obtained by us on the lower effectiveness of helogen carriers of the aromatic series in comparison with helogen carriers of the aliphatic series, conforms with the higher thermal stability of aromatic helogen derivatives.

Thus, only at temperatures of bright-red hear (700-800 degrees centigrade) does charobensene pass into diphenyl, forming a small amount of halogen and hydrogen laides, while halogen carriers of the aliphatic series (ethyl bromide, dibromoethale) become thermally unstable near 500 degrees centigrade, i.e., at temperatures more favorable for chemical reaction with the lead and lead oxide produced in the combustion chamber from thermal decomposition of the tetraethyl lead.

Judging from the deposit formation and removal of lead from the combustion chamber, it can be said that the chloronaphthalene proves less stable thermally than chlorobenzene and is approximately equivalent to the bromobenzene. The chlorobenzene proves to be more stable thermally than bromobenzene.

The introduction into the benzene nucleus of two different halogens- bromine and chlorine--decreases its thermal stability and consequently, increases its effectiveness as a lead remover.

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Comparative Evaluation of the Effectiveness of Halogen-Carriers of Varied Chemical Composition as Removers of Lead From the Combustion Chember of the Engine

Comparative tests of gasolines, with TEL and halogen carriers of various chemical composition added, on a one-cylinder unit makes possible a comparative evaluation of the effectiveness of halogen carriers as lead removers. Comparative tests of gasolines with addition of TEL and such halogen carriers as dibromosthane and dichloroethane, containing proportions of halogen equivalent to the lead in the ethyl fluid one mole of halogen to one mole of lead), showed that deposit formation in the combustion chamber of the engine in the case of dibromosthane was half as great as in the case of the dichloroethane. Thus, the weight of the deposit on the exhaust valve falls from 1.5 grams in the case of dichloroethane, to 0.5 grams in the case of dibromosthane.

A comparison of the chemical composition of the depcits shows an increase in total lead content in the deposit obtained using dichlorosthane (72.1 and 57.5 percent), smaller lead halogen content in comparison with the deposit obtained with dibromoethane (45.00 and 54.67 percent), and at the same time twice as much lead-oxide content with dichlorosthane (35.00 and 19.40 percent). All these data show that bromine is considerably more effective as a remover of lead than chlorine.

An increase in the concentration of halogen carriers in the ethyl fluid, dibromoethane or dichloroethane, to 7.5 centimoles of halogen to 2.5 centimoles of lead in one kilogram of fuel shows a systematic improvement in removal and reduction of deposit formation in the combustion chamber of the engine. This is accompanied by an increase in lead halogen deposit, a decrease in lead oxide, and at the same time, a decrease in the amount of deposit on the exhaust valve.

It was clearly shown that continuous operation of the engine on gasoline with TEL without a halogen carrier is not possible because of the deposition of rore than 10 percent of the total quantity of lead carried into the combustion chamber with the fuel. A gradual increase in halogen concentration in the ethyl fluid decreases deposit formation of the most vital parts of the engine (exhaust valve, spark plugs, etc.), increases the output of lead halides, and improves their removal from the combustion chamber.

Thus, with an increase in dichloroethans concentration from 1.25 to 7.5 centimoles in 1 kilogram of fuel, the weight of the deposit on the exhaust valve varies from 2.35 to 7.36 grams, and the lead halogen content, from 21.50 to 76.7 percent, lead oxide content, 53.5 = 2.77 percent. During the transition from 1.25 to 3.75 - 5 centimoles DUE the indices change sharply. Minimum deposit formation on the exhaust valve takes place at 7.5 centimoles of dichloroethans.

With an increas in dibromoethane concentration from 1.25 to 7.5 centimoles, the weight of deposit on the exhaust valve changes from 1.75 to 0.2 grams, lead halogen content from 27.5 to 79 percent, lead oxide content from 45 - 2.6 percent. During the transition from 1.25 to 2.5 centimoles, all indices undergo sharp changes, while in the case of dichloroethane, such a change takes place in the transition from 1.25 to 3.75 - 5 centimoles.

Minimum deposit formation on the exhaust valve is observed with 3.75 c-moles of dibromoethane, when in the case of dichloroethane it takes place at 7.5 c-moles.

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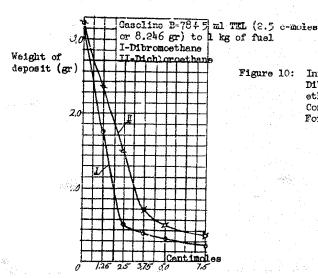


Figure 10: Influence of Change in
Dibromcethane and Dichloroethane Content in Gascline
Containing TEL, on Deposit
Formation on Exhaust Valve

Dibromoethane and dichloroethane content in moles to 1 kg of fuel

In Figure 10, the curves show change in deposit formation on the exhaust valve relative to dibromoethane (and dichloroethane) concentration in the gasoline. From these curves it follows that, in relation to deposit formation on a most vital part of the motor—the exhaust valve—an ethyl fluid containing 2.5 centimoles of dibromoethane gives approximately one third as much deposit as ethyl fluid containing an equivalent amount of dichloroethane (0.5 and 1.5 grams).

The influence of accumulation of halogen atoms in the molecule, leading to a decrease in thermal stability of the halogen carrier, was investigated with brome and chlore derivatives of the alignatic series. It was shown that ethyl bromide, in relation to deposit formation and removal of lead, is somewhat less satisfactory than dibromeethene.

Thus, on ethyl bromide the deposit from the exhaust valve and lead deposited in the combustion chamber proved to be 0.40 grams and 2.98 percent, and on dibromoethane, 0.36 grams and 2.4 percent. The effectiveness of dibromoethane and tetrabromoethane as lead ramovers proved to be practically the same.

We also failed to notice any great difference, in relation to deposit formation and removal of lead, between dichloroethane, chloroform, and carbon tetrachloride. All of these halogen carriers showed practically the same effect as lead removers. Thus, it was not possible for us to notice essential differences in the behavior of dichloroethane and carbon tetrachloride.

A comparative study of halogen carriers of the aromatic series—chlorobanzene, chloro-bromobenzene, and G-monochloronaphthalene—showed that all of these halogen carriers were considerably less effective than dibromoethene—halogen carrier of standard ethyl fluid. Thus, for example, in relation to deposit formation on the exhaust valve, chlorobenzene (1.82 grams) is much less satisfactory than dibromoethene (0.35 grams).

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carriers of the aromatic series in comparison with helogen carriers of the alignetic series conforms with the higher thermal stability of aromatic balogen derivatives. Thus, chlorobenzene, only at 700 - 800 'egreco's transforms into diphenyl, giving off in the process a small amount of free helogen and hydrogen haliles, while halogen carriers of the alighatic series (ethyl bromide, dibromothere) become thermally estable near 500 degrees centigrade; i.e., at temperatures most favorable for charlest reaction with the lead forming in the combustion chamber, from thermal decomposition of the tetraethyl lead.

It is necessary to note that X-mono-chloronaphthalane proved to be a fairly effective last remover although in this respect it is inferior to dibromoethane. Chloronaphthalane was approximately 20 percent more effective than chlorobenzene, and was practically equivalent to bromobenzene. It was shown that halogen carriers, containing a benzene nucleus with two halogens, chlorine and bromine, proved to be somewhat more effective for lead removal than halogen carriers such as chlorobenzene or bromobenzene with only one halogen in the benzene nucleus.

G. Conclusions

1. The laboratory method developed for evaluating the effectiveness of halogen cerriers of various chemical composition and the removal of antiknock compounds from the combustion chember of the engine approximates the operating conditions of circuraft engines. A comparative evaluation of entilknock compounds and halogen cerriers was cerried out under determined conditions on a one-cyl'nder waukesha engine by means of burning gasoline with the specific substances added and by a physicochemical analysis of the solid products of combustion.

The comparative method of evaluation herein developed requires a small amount of material and few facilities. However, the engine method developed cannot be recommended for the evaluation of lead removers very close to each other in their effectiveness. For the evaluation of similar halogen-carriers, a more accurate laboratory method is required.

- 2. It was shown that the removal of lead from the combustion chamber of the engine proves to be fairly high (90 percent) even without the addition of special halogen carriers to the gasoline.
- 3. It was shown that the removal of lead from the combustion chamber of the engine improves proportionally with an increase of halogen carrier concentration in the ethyl fluid.
- 4. It was shown that brome derivatives of the alighetic series (dibromoethene) give one-third the derest on the exhaust valve of the engine that chlore derivatives of the seme series give (dichloroethene).
- 5. It was shown the removal of lead from the combustion chamber of the engine varies depending on the degree of substitution of bromine for the hydrogen atoms of the hydrocarbon molecule. Ethyl bromide proved to be somewhat less effective as a remover than bromoethane and tetrahromoethane. The latter two halogen carriers, for operating conditions of a one-cylinder engine, possess practically the same effectiveness as lead removers.
- 6. It was shown that, in a one-cylinder engine the chlorine compounds of the alighetic series--dichloroethane, chloroform, and carbor tetrachloride--have practically the same effectiveness as lead removers.
- 7. It was shown that halogen carriers of the aliphatic series were more effective lead removers than halogen carriers of the eromatic series.
- 8. It was shown, on the basis of the small number of test substances of the aromatic series, that chlorobenzene was inferior as a remover of TEL, to bromobenzene, and that C -monochloronaphthalene was a better remover than the first two

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helogen carriers. It was shown that the combination in the aromatic nucleus of two halogens--bromine and chlorine (para-bromo-chlorobenzene)--proved to have a beneficial action on the removal of lead.

9. It was shown that the most effective helogen carriers of lead can be only those whose thermal decomposition takes place as close as possible to the conditions necessary for tetraethyl lead decomposition. The effectiveness of helogen carriers decreases in proportion to the increase of their decomposition temperature. A knowledge of these rules will permit a more accurate approach to the search for new, more effective lead removers.

The foregoing report was originated under the auspices of the Ministry of the Aviation Industry, Central Order of Lenin Scientific Research Institute of Aircraft Engine Construction imeni F. I. Baranov, and published as one of the scries of "Transactions of the Central Institute of Aircraft Engine Construction (TsIAM),"
No 112, by Defense Publishers, in 1946.

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II

". . . a mixture of the so-called 'aviation component' (Russian 'aviakomponent') of the straight-run gasoline fraction obtained from the best petroleums with aromatic components (benzol, pyrobenzol, sometimes campol.)"

Describing a method for obtaining B-78 gasoline with physicochemical properties conforming to GOST 1012-44.

Below is extracted from an article, "investigation of Variously Prepared Commercial Aviation Gagolines in Waukesha Engine by C. F. R. Method with Supercharging", by D. Ya. Kolomatekiy, and A. A. Deryabin in "aftyanova Khozyayatvo, No 5, 1946. (Cf. Chemical Abstracts, Vol 41, No 3, 851a)

III

Tetraethyl Lead - GOST 2657-44

Physical-Chemical Indexes

| Specific gravity df0 minimum | 1.570 |
|--|---------|
| Tetraethyl lead content in \$ by weight- minimum | 92.0 |
| Ethyl Chloride content, \$ by weight maximum | 0.6 |
| Reaction | neutral |

These data were taken from the book <u>Technical Standards for Petroleum Products</u>, issued by the <u>Technical Council for Rationalization</u> of <u>Petroleum of the Main Administration</u> of <u>National Petroleum Supply</u>, published by the <u>State publishers</u> of <u>Technical Literature</u> on <u>Fuel</u>, <u>Moscow-Leningrad</u>, 1946.

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